70627

QUALITY ASSURANCE PROJECT PLAN (QAPP) FOR

PAGEL'S PIT LANDFILL
(Winnebago Reclamation Landfill)
Remedial Investigation/Feasibility Study

Prepared By:

Warzyn Engineering Inc.
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APPROVALS:

	DATE:
Representative PRP Steering Committee	1-30-88
Warzyn Site Project Manager	1/25/88
Warzyn Quality Assurance Officer	1-25-88
EPA Remedial Project Manager	
EPA Region V Quality Assurance Officer	

The following summarizes modifications made to attached portions of the Pagel's Pit Landfill RI/FS Quality Assurance Project Plan and locations for insertion into the document dated December 1987.

Page 15 of 30 - Default insolation and growing season data for the HELP model will be derived from records for Des Moines, Iowa, rather than St. Cloud, Minnesota. Replaces page 15 of 30 in Section 3.

Page 17 of 30 - Activity 3B.4 has been rewritten to clarify that samples taken from the private well will not be filtered prior to analysis. Replaces page 17 of 30 in Section 3.

Page 18 of 30 - Subtask 3E has been modified to state that air samples will be collected and that collection will be attempted during worst case conditions, if possible. Replaces page 18 of 30 in Section 3.

Table 3 - Warzyn has replaced Hazleton as the performing laboratory for analysis of arsenic, cadmium and barium. Replaces Table 3 in Section 3.

Page 20 of 30 - Performance audit responsibility has been changed from QAO to CPMS, CRL. Replaces page 20 of 30 in Section 4.

CLP Target Compound List - Compound number 10 has been changed to Total-1,2-Dichloroethene. Replaces page 1 of Appendix A.

Page 18, Sampling Plan - Text deleted from the bottom of the page has been reinserted. Replaces page 18 of Appendix B.

Analytical Methods for Phenolics and Arsenic - Methods have been modified to include a lower concentration standard. The digestion method for Phenolics includes an example matrix spike recovery calculation. Replaces methods for Phenolics and Arsenic in Appendix D.

RCW/jpl/DWH
[jpl-101-26]

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APPROVALS:	DATE:
Representative PRP Steering Committee	
James A. Hall /dmh Warzyn Site Project Manager	1/28/88
Richard L. Mauer Warzyn Quality Assurance Officer	1.28.88
EPA Remedial Project Manager	
EPA Region V Quality Assurance Officer	

The HELP model analysis will be performed using available local climatological data. Daily precipitation data were obtained from National Oceanic and Atmospheric Administration (NOAA) for the period 1952 to the present for the Rockford Reporting Station. Editing of data to remove years containing substantial missing data will occur. Average monthly temperature data was also obtained from NOAA records for Rockford. Insolation and growing season data will be taken from the records for Des Moines, Iowa contained within the HELP model default data files. Selection of soil characteristics will be taken from the 20 sets of default soils data contained in the HELP model, based upon on-site soil classifications, and on test data for the clay barrier soils.

The HELP model analysis for the 6 inch topsoil/24 inch clay cover for closed areas and an analysis using 6 inches of sand cover for the active areas will be made using the layering and soils characteristics described in the proceeding paragraph. A sensitivity analysis will be performed to evaluate the effects of differences in rooting depths, vegetation density and competency of the clay barrier on predicted percolation rates.

Activity 3B.1 - Groundwater Monitoring System

Fifteen additional wells will be installed to supplement the existing groundwater monitoring network. At each location where a single well or nest of wells is installed, soil from the deepest boring will be sampled at five foot intervals to its terminus. Samples will be retained by Warzyn or the PRPs for inspection for a period of 1 year. No chemical analyses of these samples are planned.

Activity 3B.4 - Groundwater Quality Monitoring

A maximum of four rounds of monitoring are proposed. The initial two rounds will include 43 monitoring well samples and 1 private well sample. Samples for metals analysis will be filtered except for those from the private well which will not be filtered prior to analysis. A reduced number of samples is anticipated in rounds three and four (a minimum of 15 monitoring wells and 1 private well). For rounds three and four, samples for metals analysis will be filtered except for those from the private well which will not be filtered.

Subtask 3C - Sediment Sample Collection

Stream sediment samples will be collected from Killbuck Creek at five locations in order to evaluate the nature and extent of contamination that may be present. One upstream location and four downstream locations will be sampled. Sampling will proceed from furthest downstream location to furthest upstream location to minimize contamination from sediments disturbed in the sampling process.

Subtask 3D - Surface Water Investigations

Surface water samples will be collected at sediment sampling sites on two occasions. Sampling will be concurrent with first round groundwater sampling, and if needed, concurrent with third round groundwater sampling.

Subtask 3E - Air Monitoring

Air sampling will be conducted at three downwind and one upwind locations at property boundaries in order to obtain background data and evaluate potential impacts to downwind receptors. Samples will be collected during a time period that appears to exhibit "worst case" conditions if possible.

Weather data will be collected concurrent with air monitoring. Weather-related information will be collected using the TERRA 8 datalogger. The TERRA 8 datalogger is a fully programmable computer/data collection system that can acquire weather-related data from a variety of sensing transducers for extended periods of time. The TERRA 8 is programmable and data collected is recorded in a non-volatile RAM memory for download to the user via cassette.

The TERRA 8 is housed in a weather proof box and, when established in the field, requires minimal maintenance. Weather data is collected via specific sensing transducers, which sense and monitor information and relay it back to the TERRA 8. The information is read into the datalogger according to specific tailer-made programs by the user or by the Standard Users Program, which is company-installed into the datalogger. There are a variety of sensing transducers, i.e., dry-bulb temperature, wind speed, wind direction, relative humidity, etc. Once data has been acquired, it is kept in the TERRA 8's non-volatile RAM memory for download to the user via cassette. Data collected and stored in memory may be withdrawn with various programs that allow data files to be read from cassette tape for analysis.

4.4 SPECIALIZED RESPONSIBILITIES FOR LABORATORY SERVICES

- Hazleton Data

  - analytical protocol specified - Warzyn Engineering
  - review of analytical specifications - U.S. EPA, Region V, QA Office
  - review and approval of performing laboratory - U.S. EPA, Region V, QA Office

  - internal QA/QC - Hazleton staff
  - final data review and validation - Warzyn Engineerng
  - review of tentatively identified compounds and assessment of need for confirmation - Warzyn Engineering

- Warzyn Data

  - review of analytical specifications - U.S. EPA, Region V, QA Office
  - review and approval of performing laboratory - U.S. EPA, Region V, QA Office
  - internal QA/QC - Warzyn Engineering
  - final data review and validation - Warzyn Engineering

4.5 QUALITY ASSURANCE

- Overall QA Responsibility - Warzyn Quality Assurance Officer (QAO)

- Warzyn subcontracted activities - Warzyn Engineering

- Review of QAPP - U.S. EPA, Region V, QA Office

- Field analyses - Warzyn Engineering

4.6 PERFORMANCE AND SYSTEMS AUDITS

- Field Operations - QAO, Warzyn Engineering

- Analytical Laboratories - U.S. EPA, Region V, CPMS, CRL

- Evidence Audit - QAO, Warzyn Engineering

A project organizational chart is shown in Drawing C 12660-A1.

TABLE 3
SAMPLE TYPES AND ESTIMATED SAMPLE NUMBERS
PAGEL'S PIT LANDFILL

(1) MATRIX	(2) LAB	NO. OF SAMPLES	FIELD DUPLICATES	(3) FIELD BLANKS	(4) MATRIX SPK/DUP	(5) GC/MS CONFIRM	(6) TOTAL SAMPLES	(7) TEST PARAMETERS
GROUNDWATER								
ROUND 1	HAZLETON	44	5	3	6	5	63	VOLATILES HAZLETON'S METHOD
	HAZLETON	17	2	1	2		22	EPA HSL BNA'S AND PEST/PCB'S
	HAZLETON	17	2	1			20	EPA HSL METALS, DISSOLVED
	WARZYN	17	2	1			20	CYANIDE
	WARZYN	44	5	3			52	PHENOL
	WARZYN	44	5	3			52	ALKALINITY, CHLORIDE
	WARZYN	27	3	2			32	METALS, DISSOLVED (As, Ba, Cd)(8)
ROUND 2	HAZLETON	44	5	3	6	5	63	VOLATILES HAZLETON'S METHOD
	WARZYN	44	5	3			52	PHENOL
	WARZYN	44	5	3			52	ALKALINITY, CHLORIDES
	WARZYN	44	5	3			52	METALS, DISSOLVED (As, Ba, Cd)(8)
ROUND 3 (optional)	HAZLETON	16	2	1	2		21	VOLATILES HAZLETON'S METHOD
	WARZYN	16	2	1			19	PHENOL
	WARZYN	16	2	1			19	ALKALINITY, CHLORIDES
	WARZYN	16	2	1			19	METALS, DISSOLVED (As, Ba, Cd)(8)
ROUND 4 (optional)	HAZLETON	16	2	1	2		21	VOLATILES HAZLETON'S METHOD
	WARZYN	16	2	1			19	PHENOL
	WARZYN	16	2	1			19	ALKALINITY, CHLORIDES
	WARZYN	16	2	1			19	METALS, DISSOLVED (As, Ba, Cd)(8)
LEACHATE								
ROUND 1	HAZLETON	6	1	1	2	1	11	VOLATILES HAZLETON'S METHOD
	HAZLETON	6	1	1	2		10	EPA HSL BNA'S AND PEST/PCB'S
	HAZLETON	6	1	1			8	EPA HSL METALS, TOTAL
	WARZYN	6	1	1			8	CYANIDE
	WARZYN	6	1	1			8	PHENOL
	WARZYN	6	1	1			8	ALKALINITY, CHLORIDE
ROUND 2	HAZLETON	6	1	1	2	1	11	VOLATILES HAZLETON'S METHOD
	WARZYN	6	1	1			8	PHENOL
	WARZYN	6	1	1			8	ALKALINITY, CHLORIDES
	WARZYN	6	1	1			8	METALS, TOTAL (As, Ba, Cd)
ROUND 3	HAZLETON	6	1	1	2	1	11	VOLATILES HAZLETON'S METHOD
	WARZYN	6	1	1			8	PHENOL
	WARZYN	6	1	1			8	ALKALINITY, CHLORIDES
	WARZYN	6	1	1			8	METALS, TOTAL (As, Ba, Cd)
ROUND 4	HAZLETON	6	1	1	2	1	11	VOLATILES HAZLETON'S METHOD
	WARZYN	6	1	1			8	PHENOL
	WARZYN	6	1	1		"	8	ALKALINITY, CHLORIDES
	WARZYN	6	1	1			8	METALS, TOTAL (As, Ba, Cd)

TABLE 3
SAMPLE TYPES AND ESTIMATED SAMPLE NUMBERS
PAGEL'S PIT LANDFILL
(Continued)

(1) MATRIX	(2) LAB	NO. OF SAMPLES	FIELD DUPLICATES	(3) FIELD BLANKS	(4) MATRIX SPK/DUP	(5) GC/MS CONFIRM	(6) TOTAL SAMPLES	(7) TEST PARAMETERS
ROUND 5	HAZLETON	6	1	1	2	1	11	VOLATILES HAZLETON'S METHOD
	WARZYN	6	1	1			8	PHENOL
	WARZYN	6	1	1			8	ALKALINITY, CHLORIDES
	WARZYN	6	1	1			8	METALS, TOTAL (As, Ba, Cd)
SURFACE WATER								
ROUND 1	HAZLETON	5	1	1	2	1	10	VOLATILES HAZLETON'S METHOD
	WARZYN	5	1	1			7	PHENOL
	WARZYN	5	1	1			7	ALKALINITY, CHLORIDES
	WARZYN	5	1	1			7	METALS, TOTAL (As, Ba, Cd)
ROUND 2	HAZLETON	5	1	1	2	1	10	VOLATILES HAZLETON'S METHOD
	WARZYN	5	1	1			7	PHENOL
	WARZYN	5	1	1			7	ALKALINITY, CHLORIDES
	WARZYN	5	1	1			7	METALS, TOTAL (As, Ba, Cd)
SEDIMENT								
	HAZLETON	5	1				6	EPA HSL VOLATILES
	HAZLETON	5	1				6	EPA HSL BNA'S AND PEST/PCB'S
	HAZLETON	5	1				6	EPA HSL METALS
	WARZYN	5	1				6	CYANIDE
AIR								
	RADIAN	4	3	1			8	VOLATILES

(1) All samples are considered low concentration samples
(2) Hazleton = Hazleton Laboratories America
3301 Kinsman Boulevard
Madison, Wisconsin 53704
Warzyn = Warzyn Engineering Inc.
One Science Court
University Research Park
Madison, Wisconsin 53705
Radian = Radian Corporation
Progess Center
3200 E. Chapel Hill Rd/Nelson Hwy.
P.O. Box 13000
Research Triangle Park, North Carolina
(3) A trip blank for volatiles will be included with each shipment.
(4) Sample numbers reflect extra volume required for matrix spike/matrix spike duplicate analyses.
(5) Ten percent of all volatiles analyses by Hazleton's method will be confirmed by GC/MS analysis.
(6) Sample totals do not include trip blanks for volatiles.
(7) See Appendix A for CLP TCL list. See Appendix D for analyte list for volatiles analysis by Hazleton's method. See Appendix F for analyte list for volatiles analysis of air samples using Radian's method.
(8) Groundwater samples from private wells will not be filtered

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The remaining 27 wells to be sampled in Round 1 will be analyzed for the following:

- VOC's using Hazleton's test procedure; 10% confirmation by Gas Chromatography Mass Spectroscopy (GC/MS).
- Total Phenolics
- Field pH
- Field specific conductance
- Total alkalinity
- Chlorides
- Arsenic (Dissolved for monitoring wells, total for residential well)
- Barium (Dissolved for monitoring wells, total for residential well)
- Cadmium (Dissolved for monitoring wells, total for residential well)

The second round sampling locations will be identical to the first round.

Additional parameters may be added to the above list after mutual agreement by Pagel's Pit Landfill PRPs, U.S. EPA and Warzyn if it appears other parameters are important to defining impacts from Pagel's Pit Landfill.

The analyses will be for the parameters listed:

- VOC's using Hazleton's test procedure; 10% confirmation by Gas Chromatography Mass Spectroscopy (GC/MS).
- Total Phenolics
- Field pH
- Field specific conductance
- Total alkalinity
- Chlorides
- Arsenic (Dissolved for monitoring wells, total for residential well)
- Barium (Dissolved for monitoring wells, total for residential well)
- Cadmium (Dissolved for monitoring wells, total for residential well)

Samples collected in Rounds 3 and 4 (if necessary) will be analyzed for the same parameters analyzed in Round 2.

Sediment

The stream sediment samples will be analyzed for HSL organic and metals listed in Appendix A using CLP protocols. Total cyanide will be determined by Warzyn using the method described in Appendix D.

CLP TARGET COMPOUND LIST
AND DETECTION LIMITS*

	Volatiles	CAS Number	Detection Limits(1) Low Water(2) ug/l	Low Soil Sediment(3) ug/kg
1.	Chloromethane	74-87-3	10	10
2.	Bromomethane	74-83-9	10	10
3.	Vinyl Chloride	75-01-4	10	10
4.	Chloroethane	75-00-3	10	10
5.	Methylene Chloride	75-09-2	5	5
6.	Acetone	67-64-1	10	10
7.	Carbon Disulfide	75-15-0	5	5
8.	1,1-Dichloroethene	75-35-4	5	5
9.	1,1-Dichloroethane	75-35-3	5	5
10.	Total-1,2-Dichloroethene	156-60-5	5	5
11.	Chloroform	67-66-3	5	5
12.	1,2-Dichloroethane	107-06-2	5	5
13.	2-Butanone	78-93-3	10	10
14.	1,1,1-Trichloroethane	71-55-6	5	5
15.	Carbon Tetrachloride	56-23-5	5	5
16.	Vinyl Acetate	108-05-4	10	10
17.	Bromodichloromethane	75-27-4	5	5
18.	1,1,,2,2-Tetrachloroethane	79-34-5	5	5
19.	1,2-Dichloropropane	78-87-5	5	5
20.	trans-1,3-Dichloropropene	10061-02-6	5	5
21.	Trichloroethene	79-01-6	5	5
22.	Dibromocloromethane	124-48-1	5	5
23.	1,1,2-Trichloroethane	79-00-5	5	5
24.	Benzene	71-43-2	5	5
25.	cis-1,3-Dichloropropene	10061-01-5	5	5
26.	2-Chloroethyl Vinyl Ether	110-75-8	10	10
27.	Bromoform	75-25-2	5	5
28.	2-Hexanone	591-78-6	10	10
29.	4-Methyl-2-pentanone	108-10-1	10	10
30.	Tetrachloroethene	127-18-4	5	5
31.	Toluene	108-88-3	5	5
32.	Chlorobenzene	108-90-7	5	5
33.	Ethyl Benzene	100-41-4	5	5
34.	Styrene	100-42-5	5	5
35.	Total Xylenes		5	5

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TOTAL PHENOLICS - Distillation

Scope and Application: This method is applicable to the analysis of drinking water, surface water, domestic and industrial wastewater and solids.

Method: Automated 4AAP with distillation.

Reference: EPA 1983, Method 420.2.

Detection Limit: 0.005 mg/L

Optimum Range: 0.005 - 0.5 mg/L

Sample Handling: Aqueous samples are to be collected in a 500 mL glass container with a teflon-lined cap. Preserve to pH<2 with sulfuric acid and refrigerate at 4°C. Non-aqueous samples are to be collected in a glass container and refrigerated at 4°C. Sample should be analyzed within 28 days of collection.

Reagents and Apparatus:

1. Buffer solution
2. Phenol
3. Copper sulfate
4. Glass distillation apparatus
5. 500 mL distillation flasks
6. 500 mL erlenmeyer flasks
7. Glass funnel
8. 250 mL graduated cylinder
9. Lachat Instrument
10. Milli-Q water
11. Sodium hydroxide
12. Concentrated sulfuric acid
13. Ammonium chloride
14. Ammonium hydroxide
15. Phenol manifold.

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise noted.)

1. Buffer solution: Dissolve 67.6 g NH_4Cl in 572 mL conc. NH_4OH and dilute to 1000 mL in a volumetric flask with Milli-Q water.

2. Stock phenol solution (1000 mg/L): Dissolve 1.000 g phenol in freshly boiled and cooled Milli-Q water in a volumetric flask and dilute to 1 liter.

3. Working phenol solution A (10 mg/L): Dilute 10 mL of stock phenol solution to 1 liter with Milli-Q water in a volumetric flask. (Prepare fresh daily!)

4. Copper sulfate solution: Dissolve 100 g $CuSO_4 \cdot 5H_2O$ in Milli-Q water and dilute to 1 liter in a volumetric flask.

5. NaOH (1N): Dissolve 40 g NaOH in Milli-Q water in a 1 liter volumetric flask, dilute to the mark.

6. NaOH (6N): Dissolve 240 g NaOH in Milli-Q water in a 1 liter volumetric flask, dilute to the mark.

7. H_2SO_4 (1N): Dilute 28 mL of concentrated H_2SO_4 with 500 mL Milli-Q water in a 1 liter volumetric flask, and dilute to the mark.

Notes:

1. Interferences:

   a) Oxidizing agents such as chlorine interfere. Add excess ferrous ammonium sulfate immediately after sampling to remove interference. If chlorine is not removed, the phenols may be partially oxidized and results will be low.

   b) Sulfur compounds interfere. Interferences are eliminated by acidifying the sample to a pH of less <4 with H_3PO_4 or H_2SO_4 and aerating briefly by stirring and adding $CuSO_4$.

2. If the solutions do not remain a blue to green color after the addition of the $CuSO_4$, or lose the color upon heating, additional $CuSO_4$ is required. Record the additional amount of $CuSO_4$ required on the data sheet.

Procedure:

1. All glassware is to be soap and water washed, tap rinsed and Milli-Q water rinsed prior to analysis. Glassware which is difficult as stated can be soaked in a chromate solution for a few hours and then washed as usual.

2. For aqueous samples: Using a 250 mL graduated cylinder, place 250 mL of preserved sample into a distillation flask.

   For solid samples: Weigh out 0.5-1.0 g and place in a distillation flask. Add 250 mL Milli-Q water.

To Spike samples: Pipet 2.5 mL of 10 mg/L working phenol solution A into 250 mL of sample. This corresponds to a 0.1 mg/L spike.

3. Adjust the sample to a pH 4 ± 0.1, using 1N H_2SO_4 or 6N NaOH. Add 5 mL $CuSO_4$ solution. Add several of boiling chips to the flask.

4. Set flask on mantle, connect to condenser, and place a labeled 250 mL volumetric flask under tip of condenser. Turn the mantle to medium-high heat.

5. Distill about 200 mL of sample. Stop the distillation, and when boiling ceases, add about 50 mL of warm Milli-Q water (warm water is required to prevent the flask from cracking.) to the flask. Resume distillation until 250 mL have been collected.

6. If the distillate is turbid, filter through a prewashed 0.45 micron membrane filter.

7. Distillates are ready for analysis. Proceed with Lachat SOP# PhenAA for automated color development.

Quality Control:

1. The standards do not need to be carried through the distillation procedure.

2. A reagent blank is to be analyzed with each set of samples. This blank is to be carried through the distillation step and is used as a check for contamination.

3. A quality control calibration standard of 0.40 mg/L (which has been carried through the distillation step) is to be analyzed with each run. The last sample analyzed in the run is to be the calibration standard. These standards must be within the acceptable ranges or the samples run after the last acceptable check standard are to be reanalyzed. Record the calibration standards in the quality control book. The confidence limits are noted in the quality control book.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Duplicates are to be averaged. Spike recoveries and duplicate results are to be within acceptable ranges.

Calculation:

1. Calculate with Lachat QuikChem software, in the concentration mode, using the IBM XT computer.

2. To calculate the spike:

$$\% \text{ recovery} = \frac{[\text{Total mL}][\text{Conc. of}] \quad [\text{ml SA}][\text{Conc. of}]}{[\text{SA + STD}][\text{ spike }] - [\text{ used}][\text{ SA }]} \times 100$$
$$(\text{mL STD used})(\text{Conc. STD})$$

Where:

1. Total mL SA + STD = mL sample used plus mL standard used for the spiked sample.

2. Conc. of Spike = Measured concentration (in mg/L) of the spiked sample.

3. mL SA used = mL sample used in the spiked sample.

4. Conc. of SA = Measured concentration (in mg/L) of the sample itself.

5. mL STD used = mL standard used in the spiked sample.

6. Conc. STD = Concentration (in mg/L) of the standard used for the spiked sample.

EXAMPLE:

A technician was to determine the % recovery of a chloride spike. The concentration of chloride in the sample was determined to be 116 mg/L. The analyst used 10 mL of sample and 5 mL of the 886.5 mg/L chloride standard for the spike. The measured concentration of the spiked sample was determined to be 361 mg/L chloride. The % recovery was calculated as follows:

Total mL SA + STD = 15 mL
Conc. of Spike - 361 mg/L
mL SA used = 10 mL
Conc. of SA = 116 mg/L
mL STD used = 5 mL
Conc. STD = 886.5 mg/L

$$\frac{[15]\ [361] - [10]\ [116]}{[5]\ [886.5]} \times 100 = 96\% \text{ recovery}$$

TOTAL PHENOLICS - AUTOANALYZER

Scope and Application: This method is applicable to distilled groundwater, drinking water, wastewater, sediments and soils. All samples must be distilled prior to analysis with the autoanalyzer.

Reference: EPA, 1983, Method 420.2

Lachat Instruments, 1986, Method 10-210-00-1-A
Standard Methods, 16th Edition, pages 556-561

Detection Limit: 0.005 mg/L

Optimum Concentration Range: 0.005 - 0.500 mg/L

Sample Handling: Samples should be capped and refrigerated at 4°C after distillation.

Instrument Conditions:

1. Pump speed: 35
2. Cycle period: 40 seconds
3. Load period: 20 seconds
4. Inject period: 20 seconds
5. Inject to start of peak period: 14 seconds
6. Inject to end of peak period: 40 seconds
7. Gain: 200x10 (use 10x gain)
8. Zero: 350
9. Interference filter: 500 nm
10. Sample loop: 120 cm
11. Standards for standard curve: 0, 0.01, 0.050, 0.100, 0.200, 0.500 mg/L

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise noted.)

1. Degassed Milli-Q water - 2 options:

   a. Boil Milli-Q water vigorously for 5 minutes. Cool and store in cubitainer.

   b. Bubble helium, using the fritted gas dispersion tube, through the Milli-Q water. (15 min/20 L.) Store in cubitainer.

- Color will interfere, dilute the sample and spike the sample to confirm the quality of the result.

- Turbidity will interfere. If present, filter through a 0.45 micron membrane filter prior to analysis on Lachat System.

- A peak with a shoulder indicates an interference. If this occurs, dilute the sample. If the problem still exists, redistill the sample and it's spike at a dilution.

System Operation:

1. Refer to "Auto Analyzer Operation Start-up procedure". (SOP# LAA-section A.)

2. Spikes will be distilled at a level of 0.4 mg/L. The calibration check standard is 0.200 mg/L (D).

3. Analyze a blank and a standard at the beginning of each run. An EPA standard will only be analyzed monthly due to the inavailability of outside standards.

4. If a sample and spike are overrange:

   - Dilute the sample and spike if dilution is $\leq$ 1:5. The distilled standard will still be detectable.
   - Dilute the sample and do a manual spike if dilution is > 1:5.

5. Refer to Auto Analyzer shut-down proceduere. (SOP# LAA-section B.)

Quality Control:

1. Establish a standard curve with the standards listed above. Record the check standard in the check book. The concentration should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check reagents, pump tubing, etc.).

2. A quality control calibration standard of 0.200 mg/L is to be analyzed, at a minimum, after every 10 samples. If less than 10 samples are analyzed, a calibration standard is still required. The last sample analyzed in the run is to be the calibration standard. These standards must be within the acceptable ranges or the samples run after the last acceptable check standard are to be reanalyzed. Record the calibration standards in the quality control book. The confidence limits are noted in the quality control book.

2. 4-aminoantipyrine color reagent: In a 250 mL volumetric flask, dissolve 0.16 g of 4-aminoantipyrine ($CH_3NN(C_6H_5)COC(HN_2)\cdot CCH_3$), in approximately 150 mL of Milli-Q water and dilute to 250 mL. Prepare fresh daily.

3. Buffered potassium ferricyande (pH 10.3): In a 1 liter volumetric flask, dissolve 2.0 g potassium ferricyanide ($K_3Fe(CN)_6$), 3.1 g boric acid (H_3BO_3) and 3.75 g potassium chloride (KCl) in 800 mL of water. Add 46.8 mL of 1 M sodium hydroxide and dilute to the mark. Filter through a 0.45 micron membrane filter before each use. Stable for one month, if refrigerated.

4. Stock Phenol (1000 mg/L): In a 1 liter volumetric flask, dissolve 1.00 g of phenol in 500 mL of Milli-Q water. Add 2 mL concentrated H_2SO_4 as a preservative. Dilute to 1000 mL. (1.0 mL = 1.0 mg phenol).

5. Working Stock Standard (10.0 mg/L): In a 500 mL volumetric flask, pipet 5.0 mL of the stock phenol and dilute to the mark with Milli-Q water. (Prepare fresh daily!)

6. Standards (Prepare fresh daily!)

Concentration of Standard	Letter Identifier	Volume of 10 mg/L working standard	Dilute to
0.000 mg/L	A	0.0	100 mL
0.010 mg/L	B	0.5	500 mL
0.050 mg/L	C	0.5	100 mL
0.100 mg/L	D	1.0	100 mL
0.200 mg/L	E	2.0	100 mL
0.500 mg/L	F	5.0	100 mL

Note: Computer refers to standards by letter.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum working range.

2. The gain and zero are guidelines, they must be set each day to optimize.

3. Interferences:

   - Interferences are eliminated or reduced to a minimum by the distillation procedure.

3. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Duplicates are to be averaged. Spike recoveries and duplicates are to be within acceptable ranges or trouble-shooting must be performed.

Calculations:

1. Calculate with Lachat QuikChem software, in the concentration mode, using the IBM XT computer.

Revision Date

8-18-87

9-25-87

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ARSENIC - "CLP Intent"

Method: AA - Furnace; Direct Injection

Reference: EPA 1983, Method 206.2

"Analytical Methods for Furnace Atomic Absorption Spectrophotometry", 1982, Perkin-Elmer Corporation

"Techniques in Graphite Furnace Atomic Absorption Spectrophotometry", 1985, Perkin-Elmer Corporation

Contract Laboratory Program, "Statement of Work", July, 1985

Detection Limit: 0.002 mg/L

Optimum Concentration Range: 0.002 - 0.050 mg/L

Sample Handling: Acidify with nitric acid to pH < 2. Analyzed within 6 months. All samples must be digested prior to analysis.

Instrument Conditions:

1. Arsenic electrodeless discharge lamp with lamp energy set at 8.
2. Wavelength: 193.7 nm
3. Slit Width: 0.7 Alternate
4. Mode: Peak height
5. HGA Furnace Programming:

Step 1:	130	(dry temp)	10	(ramp time)	20	(hold time)
Step 2:	1300	(char temp)	10	(ramp time)	10	(hold time)
Step 3:	2500	(atom temp)	0	(ramp time)	3	(hold time)

* Also press the read, record and stop flow buttons; enter t = 3 sec.

Step 4:	2500	(max temp)	0	(ramp time)	3	(hold time)

Press record button.

6. Sample Volume: 20 uL
7. Standards to use for curve set-up: 5.0, 10.0, 20.0, 50.0 ug/L.
8. Pyrolytic tubes must be used.

Reagent Preparation:

1. Standard Arsenic Solution (1000 ug/L Arsenic): Pipet 1.00 mL of the 1000 ppm stock arsenic solution into a 1000 mL volumetric flask, add 1/2 mL HNO_3, and dilute to the mark with Milli-Q water. Prepare fresh every month.

2. Standards (Prepare fresh every week.):

Concentration of Standard	Volume of Arsenic Standard	Dilute to
0 ug/L	0 mL of 1000 ug/L As	100 mL
5 ug/L	1/2 mL of 1000 ug/L As	100 mL
10 ug/L	1 mL of 1000 ug/L As	100 mL
20 ug/L	2 mL of 1000 ug/L As	100 mL
50 ug/L	5 mL of 1000 ug/L As	100 mL

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

3. Nickel nitrate is added as a matrix modifier to minimize volatilization losses during the drying and charring steps.

4. The use of the background correction is required.

5. High concentrations of phosphorus interfere with this procedure. The gaseous hydride method for arsenic should be used in these cases.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Injection section of this manual.

Prior to Analysis: To 5 mL of digested sample or standard, add 0.1 mL of 5% nickel nitrate solution. Mix well.

If Arsenic is to be analyzed in concentration mode, use the 20 and 50 ug/L arsenic standards and the procedures for analyzing in the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, furnace alignment, lamp alignment, graphite tube, etc.).

2. A quality control calibration standard of 20 ug/L is to be analyzed, at a minimum, after every 10 samples. If less than 10 samples are analyzed, a calibration standard is still required. The last sample analyzed in the run is to be the calibration standard. These standards must be within acceptable ranges or the samples run after the last acceptable calibration standard are to be reanalyzed. Record the calibration standard in the quality control book. The confidence limits are noted in the quality control book.

3. A digested duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a digested duplicate and spike are still required. Duplicates are to be averaged. Spike samples with a standard of twice the concentration of the sample in a 1:1 ratio of sample to standards. Spike recoveries and duplicate results are to be within acceptable ranges or the use of dilution, or method of standard additions is to be applied to reduce the interferences.

4. For every sample analyzed, an analytical spike (at the bench) must be run to verify that standard additions are not required. Criteria for standard additions are:

   a. If the spike recovery is within 85 - 115%, standard additions are not required.

   b. If the spike recovery is outside 85 - 115%, standard additions are required.

5. An EPA reference sample will be analyzed with each analysis.

Calculations:

1. Plot concentrations vs. absorbance on graph. Determine unknowns using graph, or

2. Calculate using linear regression, or

3. Calculate using the concentration mode.

Revision Date

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